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SPECIFICATIONMANUFACTURING METHOD FOR COMPOSITE MATERIALAND COMPOSITE MATERIAL OBTAINED THEREBY

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## TECHNICAL FIELD

The present invention relates to a manufacturing method for a composite material comprising two or more metals or nonmetals and compounds thereof and, more particularly, to a manufacturing method in which a dispersion material can be dispersed very homogeneously into a base material of the composite material independently of the composition of the composite material.

## 15 BACKGROUND ART

Conventionally, composite materials comprising metals or nonmetals or compounds thereof have been used in a wide variety of applications as structural materials for automobile parts, aircraft parts, etc., electrode materials, target materials for film formation, and the like. The composite material is manufactured by dispersing metals or nonmetals or compounds thereof different from a base material into the base material to control material properties so that material properties suitable for each application can be realized. The term "nonmetal" used in this specification includes hydrogen, boron, carbon, silicon, nitrogen, phosphorous, and the like, and is used as a wide concept

including antimony, bismuth, etc. which are what we call a semimetal.

As a manufacturing method for the above-described composite material, the compo-casting process and the powder metallurgy process are known. In the compo-casting process, a metal, which is used as a base material, is semi-melted, and nonmetal particles, which are used as a dispersion material, are put into the semi-melted metal and are strongly agitated, by which the nonmetal particles are compulsorily dispersed into the base metal. This process is effective in the case where the composite material is manufactured by using nonmetal particles having poor wettability with respect to the melted metal of base material. With this process, the composite material is manufactured in a semi-melting state so as to prevent separation of nonmetal particles from base metal. In the compo-casting process, however, since the semi-melted metal is in what we call a sherbet state, a defect such as a cavity is liable to occur in the obtained molded product, resulting in a tendency for gas to be involved, thereby decreasing the density of material.

In the powder metallurgy process typically represented by hot pressing, hot isostatic pressing (HIP), metal powder, which is used as a base material, and nonmetal powder, which is used as a dispersion material, are mixed with each other at a predetermined ratio, and these powders are molded and sintered to manufacture a composite material. In this process, for an easily oxidizable metal, the concentration of oxygen in the obtained composite material is high because

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the starting material is powder, so that it is sometimes difficult to control the properties of the composite material. In the powder metallurgy process, since there is restriction of powder control and mixing treatment of powder, it is 5 sometimes difficult for the dispersion material to disperse more homogeneously into the base material.

Besides the compo-casting process and the powder metallurgy process, the fusion-casting process is generally used. However, in the case where both of a base material and 10 a dispersion material are metals, and they have properties of a low melting point metal and a high melting point metal, respectively, it is very difficult to manufacture such a metal-metal composite material by, for example, the vacuum fusion process.

15 A composite material obtained by the conventional manufacturing method is described in more detail by taking a sputtering target material as an example. In recent years, when the wiring for a liquid crystal display or a semiconductor integrated circuit is formed, the wiring technology using a sputtering method using a target material 20 of composite material has been employed. In forming the wiring by sputtering, an aluminum film, which has high heat resistance and low electrical resistance, is typically used, and a target material of composite material using aluminum 25 as a base material is used to form the aluminum film.

For the aluminum film used as the wiring for a liquid crystal display or a semiconductor integrated circuit, for example, a target material of composite material in which

aluminum is used as a base material and carbon and a group  
IVa metal such as titanium are dispersed is used. This is  
because if such a target material of composite material made  
of aluminum is used, the wiring having high heat resistance  
5 and low electrical resistance can be formed, so that breakage  
of wiring caused by stress can be prevented. For this reason,  
the target material of composite material made of aluminum  
is naturally required to have a composition capable of forming  
a film meeting the requirements for the wiring  
10 characteristics. Furthermore, the target material is  
required to have less defects such as a cavity and a void,  
to have a high density, and to entrain less gas that forms  
impurities.

In the prior art described above, although a composite  
15 material can be manufactured by using aluminum as a base  
material and carbon and a group IVa metal as dispersion  
materials, it is difficult to manufacture a composite  
material that meets the requirements for the target material  
for wiring formation. Specifically, even if the  
20 aforementioned target material made of aluminum is  
manufactured by the compo-casting process, the powder  
metallurgy process, or the fusion casting process, there is  
a limit in homogeneously dispersing carbon and a group IVa  
metal in the aluminum base material, so that the obtained  
25 composite material is insufficient as a target material that  
can steadily perform wiring formation meeting the  
requirements for the practical wiring characteristics. In  
order to use a composite material as a target material, a bulk

body having a volume of some degree is needed. If a composite material for the bulk body is formed by the conventional manufacturing method, however, an internal defect such as a cavity occurs, and thus the density of bulk body tends to decrease. Also, gas and other impurities enter into the material in many cases. Therefore, even if the bulk body obtained by the conventional manufacturing method is used as a target material, it is difficult to realize steady wiring formation by sputtering.

As seen from this example of the target material, a dispersion material can be dispersed into a base material in the conventional manufacturing method for a composite material, however the dispersibility is insufficient, and an internal defect and entrance of impurities occur in the bulk body. Thus, the conventional composite material has many drawbacks to be improved. In addition to the target material, considering the case where a composite material used for other applications as a structural material for an automobile part, aircraft part, etc. or an electrode material is manufactured, in the conventional manufacturing method, it is very difficult to manufacture a composite material having various compositions generally by one manufacturing method.

The present invention has been made in view of the above situation, and accordingly an object thereof is to provide a manufacturing method for a composite material comprising two or more metals or nonmetals and compounds thereof, in which a dispersion material can be dispersed very homogeneously into a base material of the composite material,

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and which method can be used generally independently of the composition of the composite material as compared with the conventional manufacturing method.

## 5 DISCLOSURE OF THE INVENTION

To solve the above problems, the inventors earnestly conducted studies paying attention to the vapor phase epitaxy technology used for film formation. As a result, we completed a technology capable of manufacturing a composite material 10 that has not been realized by the conventional manufacturing method.

First, the present invention provides, as a first invention, a manufacturing method for a composite material in which a metal or a nonmetal or a compound thereof is used 15 as a base material, and at least one kind of metals or nonmetals or compounds thereof different from the base material is dispersed as a dispersion material, wherein a raw material for base material comprising a metal or a nonmetal or a compound thereof for forming the base material and at least one of raw materials for dispersion material comprising metals or nonmetals or compounds thereof for forming the dispersion material are evaporated simultaneously or alternately, and the evaporated particles are deposited on a substrate to form a bulk body.

25 In the first invention, the raw material for base material for forming the base material and the raw material for dispersion material for forming the dispersion material are made evaporated particles by what we call the physical

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vapor deposition process (PVD process), and the evaporated particles are deposited on the substrate to form the bulk body. According to the first invention, since the raw materials for forming the base material and the dispersion material are 5 deposited as evaporated particles, unlike the conventional manufacturing method, the dispersion material is dispersed very homogeneously into the base material, so that various composite materials can be manufactured easily independently of the properties of each raw material. That is to say, even 10 a composite material of a combination of a high melting point metal and a low melting point metal can also be manufactured easily.

In this first invention, the sputtering method or the vacuum deposition method in the physical vapor deposition 15 process is preferably used. The reason for this is that in these methods, evaporated particles are produced from each raw material at relatively high rate, so that a bulk body having a predetermined volume can be formed easily. When the sputtering method or the vacuum deposition method is applied 20 in the first invention, since the raw material is evaporated in an atmosphere of inert gas such as argon or in a vacuum atmosphere, even an easily oxidizable raw material can be used. Therefore, the quantity of oxygen entering into the manufactured bulk body can be controlled, and the entrance 25 of gas and other impurities can be avoided to the utmost. Further, a bulk body of composite material having far less internal defects can be manufactured.

PROSPECTUS  
OF THE INVENTION

In the first invention, the raw material for base material and the raw material for dispersion material can be evaporated simultaneously or alternately. In the case where the raw materials are evaporated simultaneously and deposited, 5 the evaporated particles of the raw material for base material and the raw material for dispersion material are deposited at random. Even in the case where the raw materials are evaporated alternately, macroscopically, a composite material in which the dispersion material is dispersed 10 homogeneously into the base material is manufactured by controlling the deposit layer of the base material and the dispersion material on the order of angstrom. Also, in the first invention, considering the fact that the bulk body is formed in a relatively short period of time, the sputtering 15 method is preferably used for the evaporation of raw materials.

Next, as a second invention, the inventors invented a manufacturing method for a composite material in which a metal or a nonmetal or a compound thereof is used as a base material, and at least one kind of metals or nonmetals or compounds 20 thereof different from the base material is dispersed as a dispersion material, wherein a raw material for evaporation comprising a metal or a nonmetal or a compound thereof for forming the base material or a metal or a nonmetal or a compound thereof for forming the dispersion material is evaporated in 25 an atmosphere of any one of a hydrocarbon gas, oxygen gas, and nitrogen gas, and the evaporated particles are deposited on a substrate to form a bulk body.

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This second invention is based on the physical vapor deposition process (PVD process) or the chemical vapor deposition process (CVD process). As the atmosphere in which the raw material for evaporation is evaporated, any one of 5 a hydrocarbon gas, oxygen gas, and nitrogen gas is selected, so that a composite material in which carbides, nitrides, or oxides are dispersed as a dispersion material very homogeneously into the base material can be manufactured. To evaporate the raw material for evaporation in the second 10 invention, the sputtering method and the vacuum deposition method in the physical vapor deposition process or an activated deposition method in the chemical vapor deposition process is preferably used.

The hydrocarbon gas used in the second invention is not 15 subject to any special restriction as to the composition thereof if it can be decomposed into carbon and hydrogen at the time of sputtering or deposition. Preferably, methane, ethane, and acetylene gas can be cited. For the atmosphere in which the raw material is evaporated in the second 20 invention, by containing an inert gas such as argon, the evaporation efficiency of raw material can be controlled.

In the second invention, a raw material for evaporation comprising a metal or a nonmetal or a compound thereof for forming the base material may be used, or a raw material for 25 evaporation containing a metal or a nonmetal or a compound thereof for forming the dispersion material in addition to the base material may be used. For example, a raw material for evaporation comprising copper for forming the base

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material and silicon for forming the dispersion material is used to produce evaporated particles in nitrogen gas by using the sputtering method, and the particles are deposited on the substrate, so that silicon and nitrogen react with each other 5 to yield stable silicon nitride. Therefore, a composite material in which silicon nitride, which is a dispersion material, is dispersed very homogeneously into copper, which is a base material, can be manufactured. Likewise, a raw material for evaporation comprising copper as the base 10 material and aluminum as the dispersion material are used to produce evaporated particles in oxygen gas by using the sputtering method, and the particles are deposited on the substrate, so that aluminum and oxygen react with each other to yield stable aluminum oxide. Therefore, a composite 15 material in which aluminum oxide, which is a dispersion material, is dispersed very homogeneously into copper, which is a base material, can be manufactured.

According to the second invention as well, a composite material that has not been realized by the conventional manufacturing method, that is, a composite material in which even a dispersion material having poor wettability with respect to the base material is dispersed very homogeneously into the base material can be manufactured. By controlling the atmosphere in which the raw material is evaporated, the 20 entrance of impurities can be restrained to the utmost, and a bulk body having far less internal defects can be manufactured. In the second invention, considering the fact 25 that the bulk body is formed in a relatively short period of

time, the sputtering method is preferably used for the evaporation of raw material.

The composite material obtained by the manufacturing methods of the first and second inventions of the present invention described above is a bulk body deposited and formed on the substrate. It is not difficult for this bulk body to be handled as a simple body, unlike what we call a film. By separating the bulk body from the substrate, the bulk body has a volume of a degree such that the bulk body itself can be handled as it is. The bulk body separated from the substrate by the manufacturing methods of the first and second inventions can be used as it is for each application, for example, as a target material.

Also, in the present invention, the bulk body obtained by the manufacturing methods of the first and second inventions is melted, mixed, and formed by casting together with the raw material for base material comprising a metal or a nonmetal or a compound thereof for forming the base material, by which the concentration of dispersion material can be controlled. In the manufacturing method of the first and second inventions, the obtained composite material of bulk body is structurally ideal in that the dispersion material is dispersed very homogeneously into the base material. However, these two manufacturing methods are based on the vapor phase epitaxy method, so that in the case of the bulk body having a larger volume, manufacturing must be performed for a long period of time, and also it is difficult to obtain a complicated bulk body. Thereupon, the bulk body

obtained by these two methods is melted, mixed, and formed by casting together with the raw material for base material to control the concentration of dispersion material, by which a composite material of a larger bulk body is manufactured.

- 5 If a mold of a predetermined shape is used at the time of casting, a composite material of a complex shape can be obtained easily.

In the case where the bulk body obtained by the first and second inventions and the raw material for base material are melted, mixed, and formed by casting, a phenomenon that the dispersion material is separated from the base material may possibly be caused as in the conventional manufacturing method. In the first and second inventions of the present invention, however, the bulk body is formed in a state in which 15 the base material and the dispersion material are dispersed into each other very finely, that is, the bulk body is formed in a state in which the dispersion material has high wettability with respect to the base material. Therefore, even if the bulk body is melted together with the raw material 20 for base material, the dispersion material is not separated from the base material. Thereupon, the composite material obtained by melting, mixing, and casting the bulk body and the raw material for base material is in a state in which the dispersion material is dispersed very homogeneously into the 25 base material. In the case where a composite material is manufactured by melting the bulk body and the raw material for base material in this way, when the bulk body is formed, the quantity of dispersion material or the quantity of raw

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material for base material to be added is controlled in advance, by which the composition of the finally obtained composite material can be controlled easily.

The temperature for melting the bulk body and the raw 5 material for base material may be determined appropriately according to the composition of composite material.

Basically, the melting may be performed at a temperature in the range from the melting point to the evaporating temperature of the bulk body. In effect, the temperature may 10 be controlled so that the bulk body becomes in a fully flowing state and the raw material for base material and the bulk body can be mixed homogeneously with each other. The atmosphere when the melting is performed is not subject to any special restriction. For the composite material in which the base 15 material or the dispersion material is easily oxidized, however, the melting is performed preferably in a vacuum atmosphere or in an atmosphere of inert gas such as argon gas. Further, when casting is performed after melting and mixing, the casting is preferably performed under a rapid 20 solidification condition. This is because if the casting is performed under a rapid solidification condition, the crystal structure of composite material is made fine, and the dispersion material is dispersed into the base material homogeneously and finely.

25 For the bulk body formed in accordance with the first and second inventions and the composite material obtained by melting, mixing, and casting that bulk body and the raw material for base material, the crystal structure thereof can

be controlled by rolling or heat treatment. The finally manufactured composite material must have properties suitable for each application. A composite material having properties suitable for each application, for example, high strength properties can be realized by controlling the crystal structure thereof by means of rolling or heat treatment. In this case, both of rolling and heat treatment may be applied, or only either of them may be applied.

In the manufacturing method for a composite material in accordance with the first and second inventions, it is preferable that the evaporated particles be deposited while the substrate is rotated. By using the rotating substrate rotated at a predetermined constant rotational speed, the deposition of evaporated particles proceeds homogeneously at all locations on the surface of the rotating substrate, so that as compared with the case where the evaporated particles are deposited on the stationary substrate, a bulk body with a more uniform composition and a uniform thickness can be formed.

Further, the substrate on which the evaporated particles are deposited is preferably made of the same material as the base material. In this case, deposited particles deposit in conformity with the substrate, so that a homogeneous crystal structure can be obtained easily. Also, in the case where a composite material is manufactured by melting, mixing, and casting the bulk body and the raw material for base material, if the material of the substrate is the same as the base material, the formed bulk body can be melted without being

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peeled off from the substrate, so that the manufacturing process can be simplified.

In the manufacturing method for a composite material in accordance with the present invention, a composite material suitable for each application can be manufactured generally independently of the composition thereof, the dispersion material is dispersed very homogeneously into the base material, the entrance of impurities is restrained, and a composite material of bulk body having no internal defect such as a cavity can be obtained. Therefore, the composite material obtained by the manufacturing method in accordance with the present invention can be practically used appropriately for each application, and can be used very suitably as a structural material for an automobile part, aircraft part, etc., an electrode material, or a target material for film formation.

Further, the composite material obtained by the manufacturing method in accordance with the present invention, in which aluminum is used as the base material and carbon is used as the dispersion material, is very suitable as a target material. As described above, an aluminum film is effectively used for the wiring for a liquid crystal display or a semiconductor integrated circuit. Conventionally, the use of a so-called mosaic-like target material in which when an aluminum film containing carbon is formed by the sputtering method, a chip etc. comprising carbon or silicon is embedded directly in an aluminum metal material has been known (Japanese Patent Laid-Open No. 2-292821). However, for such

a mosaic-like target material, problems of the heterogeneous composition of formed film and the occurrence of dust have been pointed out, and the target material of this type has not been used practically for film formation. On the other 5 hand, according to the composite material obtained by the manufacturing method in accordance with the present invention, carbon is dispersed into aluminum, which is the base material, very homogeneously and finely. Therefore, if such a composite material is used as a target material to form wiring, 10 wiring having high heat resistance and low electrical resistance can be formed steadily.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a schematic view showing a case where a bulk 15 body is formed on a stationary substrate by the sputtering method;

Fig. 2 is a schematic view showing a case where a bulk body is formed on a stationary substrate by the vacuum deposition method;

20 Fig. 3 is a schematic view showing a case where a bulk body is formed on a rotating substrate by the sputtering method;

Fig. 4 is a schematic view showing a case where a bulk body is formed on a rotating substrate by using both of the 25 sputtering method and the vacuum deposition method;

Fig. 5 is a micrograph of a cross section of a composite material having been subjected to water-cooled casting in Example 1;

Fig. 6 is a schematic view showing a case where a bulk body is formed on a stationary substrate by introducing hydrocarbon gas using the sputtering method; and

5 Fig. 7 is a schematic view showing a case where a bulk body is formed on a stationary substrate by introducing hydrocarbon gas using the deposition method.

BEST MODE FOR CARRYING OUT THE INVENTION

Preferred embodiments of a manufacturing method for a  
10 composite material in accordance with the present invention  
will be described. A first embodiment described below  
relates to a manufacturing method of the before-mentioned  
first invention, and a second embodiment relates to a  
manufacturing method of the before-mentioned second  
15 invention.

First embodiment: The first embodiment relates to a  
manufacturing method in which a bulk body is formed by  
evaporating a raw material for base material and a raw  
material for dispersion material using the sputtering method  
20 or the vacuum deposition method. Figs. 1 to 4 are schematic  
views showing various manufacturing methods of the first  
embodiment.

Fig. 1 shows a method in which a metal raw material used  
as a base material and a nonmetal raw material used as a  
25 dispersion material are evaporated by the sputtering method  
and are deposited on a plate-shaped stationary substrate. A  
plate-shaped stationary substrate 2 is installed in a chamber  
1, and a metal target 4 for base material and a nonmetal target

5 for dispersion material are provided on a respective  
substrate 3 so as to face the stationary substrate 2. The  
stationary substrate 2 and the targets 4 and 5 are connected  
to a not-shown electric power source. Also, the stationary  
5 substrate 2 is formed of a metal for base material. Although  
Fig. 1 shows a case where two targets of the metal target 4  
for base material and the target 5 for dispersion material  
are used, a plurality of targets can be provided appropriately  
according to the composition of the intended composite  
10 material.

An inert gas, for example, argon gas is introduced in  
the chamber 1, and the pressure of the gas is controlled to  
a predetermined value. Thereafter, a predetermined voltage  
is applied between the metal target 4 for base material and  
15 the stationary substrate 2 and between the nonmetal target  
5 for dispersion material and the stationary substrate 2 to  
cause a sputtering phenomenon, by which a metal for base  
material and a nonmetal for dispersion material are  
evaporated and deposited on the stationary substrate 2. The  
20 voltage may be applied to cause the sputtering phenomenon  
simultaneously on both of the targets 4 and 5, or may be applied  
to cause the sputtering phenomenon alternately. Although a  
DC 2-pole sputtering system is shown in Fig. 1, what we call  
a high-frequency sputtering system or what we call a magnetron  
25 sputtering system may be used.

By performing sputtering for a given period of time in  
this manner, a composite material of a bulk body 6 is formed  
on the stationary substrate 2. After the predetermined bulk

body 6 is formed, the bulk body 6 is removed from the stationary substrate 2 by grinding or etching the stationary substrate 2. Thereby, the simple bulk body 6 can be used in a variety of applications as a structural material, electrode material, or target material. Although this bulk body 6 can be used without being subjected to treatment, it can also be used by being subjected to rolling or heat treatment if necessary to control the crystal structure.

Also, without removing the bulk body 6 from the stationary substrate 2, the bulk body 6 and the stationary substrate 2 are heated and melted together with a base material metal having been prepared separately. By controlling the quantity of the base material metal having been prepared separately, the composition of the finally obtained composite material, that is, the concentration of the dispersion material can be determined arbitrarily. After the materials are heated to a predetermined temperature and are melted into a flowable state to some degree, they are agitated sufficiently to be mixed homogeneously, and then are formed by casting under a rapid solidification condition, by which a composite material having the intended composition and shape can be obtained. Further, if necessary, the composite material can be rolled or heat-treated to control the crystal structure.

Fig. 2 shows a method in which a metal raw material used as a base material and a nonmetal raw material used as a dispersion material are evaporated by the vacuum deposition method and are deposited on a plate-shaped stationary

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substrate. The plate-shaped stationary substrate 2 is installed in the chamber 1, and a metal deposition source 8 for base material and a nonmetal deposition source 9 for dispersion material are provided on a respective deposition 5 crucible 7 so as to face the stationary substrate 2. Both of the deposition sources 8 and 9 are connected to an electric power source, not shown. The deposition source, if being configured so that a rod-like source can be supplied continuously, is effective in achieving mass production of 10 composite material. Also, the stationary substrate 2 is formed of a metal for base material. In the case of the vacuum deposition method described with reference to Fig. 2, as in the case of Fig. 1, many deposition sources can be provided appropriately according to the composition of the intended 15 composite material.

The chamber 1 is decompressed to a predetermined pressure to produce a vacuum atmosphere. The metal deposition source 8 for base material and the nonmetal deposition source 9 for dispersion material are heated by causing electric current 20 to flow, and thereby a metal for base material and a nonmetal for dispersion material are evaporated from the deposition sources 8 and 9, respectively, and are deposited on the stationary substrate 2. By performing deposition for a given period of time in this manner, a composite material of the 25 bulk body 6 is formed on the stationary substrate 2. After the predetermined bulk body 6 is formed, as in the case of description given with reference to Fig. 1, the bulk body 6 is used as a single body, or it can be used as a composite

material in which the concentration of dispersion material has been controlled by melting, mixing, and casting the bulk body 6 together with the metal for base material. Also, if necessary, the composite material can be rolled or heat-treated to control the crystal structure.

A manufacturing method using a rotating substrate will be described with reference to Figs. 3 and 4. Fig. 3 shows a case where a composite material of a bulk body is manufactured on a rotating substrate by the sputtering method. A cylindrical rotating substrate 10 is installed in the chamber 1, and the metal target 4 for base material and the nonmetal target 5 for dispersion material are provided on the respective substrate 3 so as to face the rotating substrate 10 and to be at right angles to each other.

In this case as well, argon gas is introduced in the chamber 1, and a predetermined voltage is applied by means of a not-shown electric power source to perform sputtering, by which a metal for base material and a nonmetal for dispersion material are deposited on the side face of the cylindrical rotating substrate 10 being rotated to form a bulk body 6'. By forming the bulk body 6' using the rotating substrate in this manner, the microstructure of the bulk body 6' is made such that the metal for base material and the nonmetal for dispersion material are deposited in layers on the order of angstrom. However, viewing the whole of the bulk body 6' macroscopically, the metal for base material and the nonmetal for dispersion material have a homogeneous

composition, and the dispersion material is dispersed very finely into the base material.

As in the case of the description given with reference to Fig. 1, the bulk body 6' formed on the rotating substrate 5 can be used without being subjected to treatment as a composite material for each application, or it can be used as a composite material in which the concentration of dispersion material has been controlled by melting the bulk body 6' together with the metal for base material. Further, 10 the composite material can be rolled or heat-treated to control the crystal structure. Although Fig. 3 shows a case where two targets are used, three or more targets can be provided as a matter of course around the rotating substrate according to the composition of the intended composite 15 material. The above description with reference to Fig. 3 has been given merely to explain the case where the sputtering method is used, and a composite material of the bulk body 6' can be manufactured in the same way using the rotating substrate 10 by the vacuum deposition method. In the case 20 of the vacuum deposition method, basically, it is necessary only that the targets 4 and 5 in Fig. 3 be replaced by deposition sources, so that the detailed description thereof is omitted.

Fig. 4 shows a case where a composite material of a bulk 25 body is manufactured on a rotating substrate through use of both the sputtering method and the deposition method. The cylindrical rotating substrate 10 is installed in the chamber 1, and the metal target 4 for base material is provided on

the substrate 3 and the nonmetal deposition source 9 for dispersion material is provided in the deposition crucible 7 so that they face the rotating substrate 10 and are at right angles to each other.

5 In this case, after argon gas is introduced and the pressure thereof is controlled to a predetermined value, a metal for base material is evaporated by sputtering, and on the other hand, a nonmetal for dispersion material is heated by causing an electric current to flow until the temperature 10 thereof reaches a temperature at which the vapor pressure of the nonmetal substance is higher than the pressure in the chamber 1, whereby the nonmetal for dispersion material is evaporated. Thereby, the metal for base material and the nonmetal for dispersion material are deposited on the side 15 face of the rotating substrate 10 to form the bulk body 6'. For the formed bulk body 6', a composite material having the same construction as that described with reference to Fig. 3 can be obtained. The bulk body 6' can be handled in the same way as described with reference to Fig. 1, so that the 20 explanation is omitted.

The following is a description of examples relating to the first embodiment.

Example 1: Example 1 represents a case where an aluminum-carbon composite material is manufactured by the use of the 25 rotating substrate shown in Fig. 3 by the sputtering method. Two kinds of materials of aluminum (purity: 99.999%) used as a metal target for base material and carbon (purity: 99.9%) used as a nonmetal target for dispersion material were

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prepared. Each of the targets measures 127 mm long, 279.4 mm wide, and 10 mm thick. As a sputtering apparatus, a 3-cathode magnetron sputtering type apparatus was used, and two cathodes of the three cathodes were used. Also, an 5 octagonal cylindrical rotating substrate, which was produced by connecting the lengthwise sides of eight stainless steel plates each measuring 279 mm long and 80 mm wide to each other, was prepared. Aluminum foil (purity: 99.999%) with a thickness of 12  $\mu\text{m}$  was wound around the side face of the 10 rotating substrate, and aluminum and carbon were deposited on the aluminum foil.

The sputtering conditions were as follows: Argon gas was introduced in the chamber, the sputtering pressure was 0.87 Pa, the applied electric power was 12 kW ( $24.8 \text{ W/cm}^2$ ) for the 15 aluminum target and 4 kW ( $8.3 \text{ W/cm}^2$ ) for the carbon target, and the rotational speed of the rotating substrate was 30 rpm. Sputtering was performed for about 30 hours to form a bulk body with a thickness of 0.6 mm on the side face of the rotating substrate. The formed bulk body had a cross-sectional 20 structure in which an aluminum layer with a thickness of about 0.3  $\mu\text{m}$  and a carbon layer with a thickness of about 0.01  $\mu\text{m}$  are laminated, the thicknesses being converted from the film forming rate. The carbon concentration in the bulk body was shown to be 2.6% by weight (5.6% by atomic weight) by analysis.

25 The bulk body was melted under vacuum together with aluminum (purity: 99.999%) which had been prepared separately, by which the aluminum-carbon composition was controlled so that the carbon concentration is 0.7% by weight, and casting

was performed using a water-cooled copper mold. The composite material formed by casting was observed under a microscope. As a result, it was verified that carbon with a particle diameter of about 1 mm was dispersed into aluminum of base material in a form of Al-C( $\text{Al}_4\text{C}_3$ ) phase. Fig. 5 shows an observation result for a dispersed state of Al-C( $\text{Al}_4\text{C}_3$ ) phase for the aluminum-carbon composite material obtained in Example 1. In Fig. 5, a black portion represents the Al-C( $\text{Al}_4\text{C}_3$ ) phase.

The aluminum-carbon (0.7% by weight) composite material formed by casting as described above was formed into a sputtering target material, and an aluminum film was formed by using this target material. The aluminum film forming conditions were as follows: a DC magnetron sputtering apparatus was used, the sputtering pressure was 0.333 Pa (2.5 mTorr), and the applied electric power was 3 Watt/cm<sup>2</sup>. Under these conditions, a film with a thickness of about 3000 angstroms was formed on a glass substrate. The sputtering time required for forming a film with a thickness of about 3000 angstroms was about 100 seconds. After the film of 3000 angstroms was formed, the glass substrate was replaced, and a film was further formed. By repeating the film forming operation, film forming was performed continuously for a long period of time by using one sputtering target material. The occurrence of hillock, which represents heat resistance properties, was investigated for the film formed at the first time, the film formed when about 20 hours of sputtering time in total had elapsed, and the film formed when about 40 hours

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of sputtering time in total had elapsed. A hillock in this specification means a bump-like protrusion produced on the surface of film when the glass substrate with film is heat-treated under vacuum at a temperature of 300°C for a given period of time. As a result, it was verified that a hillock scarcely occurred on each film independently of the total sputtering time. Also, the electrical resistivity of each film was measured. As a result, it was verified that the electrical resistivity was about 5  $\mu\Omega\text{cm}$  under the optimum condition. These results exhibit very excellent wiring characteristics for a liquid crystal display and a semiconductor integrated circuit. Therefore, it was found that if the composite material obtained in Example 1 is used as a raw material for a target material, a film having excellent film characteristics can be formed steadily.

Comparative Example 1: Comparative Example 1 uses the compo-casting method. Two kilograms of aluminum (purity: 99.999%) was heated to about 700°C in a carbon crucible. After the aluminum was melted once and was then cooled to about 640°C, by which the aluminum was made in a semi-melted state (solid-liquid coexisting state). In this state, 15 g of carbon powder with an average particle size of 150  $\mu\text{m}$  was put in the aluminum melt, and was strongly agitated with an agitator. Thereafter, the aluminum was cast in a water-cooled copper mold. The obtained ingot had a plate shape measuring 200 mm long × 200 mm wide × 20 mm thick. The concentration of carbon in a portion corresponding to the bottom side of the copper mold was investigated. As a result,

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it was verified that the carbon concentration was 0.003 to 0.008% by weight, and carbon was scarcely dispersed. The ingot was observed visually, and it was verified that carbon was segregated at the upper part of the ingot. The reasons for this are presumed to be that there was hardly wetting of aluminum and carbon in the compo-casting method and that carbon was separated from aluminum and floated upward by a difference in specific gravity between aluminum and carbon.

Second embodiment: A second embodiment relates to a manufacturing method in which a raw material for evaporation is evaporated in a mixture atmosphere of a gas of any one of a hydrocarbon gas, oxygen gas, and nitrogen gas and an inert gas such as argon gas, and evaporated particles are deposited on a substrate, by which a bulk body is formed. Figs. 6 and 7 are schematic views showing various manufacturing methods of the second embodiment. Fig. 6 shows a case where the sputtering method is used, and Fig. 7 shows a case where the deposition method is used.

In the case where the sputtering method is used, as shown in Fig. 6, the plate-shaped stationary substrate 2 is installed in the chamber 1, and a target 11 for evaporation, which is formed of a metal for base material and a nonmetal for dispersion material, is provided on the substrate 3 so as to face the stationary substrate 2. The stationary substrate 2 and the target 11 for evaporation are connected to an electric power source 12. Also, the stationary substrate 2 is formed of a metal for base material. Although a DC 2-pole sputtering system is shown in Fig. 6, what we call

a high-frequency sputtering system or what we call a magnetron sputtering system can be used.

The chamber 1 is provided with an atmospheric gas inlet 13 and an atmospheric gas outlet 14. From the atmospheric gas inlet 13, an atmospheric gas produced by mixing a hydrocarbon gas such as acetylene gas and an inert gas such as argon gas is supplied, and the atmospheric gas is introduced in the chamber 1.

After the pressure in the chamber 1 is controlled to a predetermined value, a predetermined voltage is applied to cause a sputtering phenomenon, by which the metal for base material and the nonmetal for dispersion material are evaporated from the target 11 for evaporation. At this time, the hydrocarbon gas such as acetylene gas introduced in the chamber 1 decomposes into carbon and hydrogen. For example, the carbon is taken into the bulk body 6 formed on the stationary substrate 2 together with the evaporating metal for base material and nonmetal for dispersion material. Alternatively, the carbon reacts with the evaporating metal for base material or nonmetal for dispersion material to yield a stable carbide, and is taken into the bulk body 6 formed on the stationary substrate 2 in the carbide state. In this case, the quantity of the hydrocarbon gas in the atmospheric gas introduced in the chamber 1 or the voltage applied at the time of sputtering is controlled, by which the carbon concentration in a composite material formed as the bulk body 6 can be determined appropriately.

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Next, the case where the deposition method is used as shown in Fig. 7 is described. In Fig. 7, the plate-shaped stationary substrate 2 is installed in the chamber 1, and a deposition source 15 formed of a metal for base material and 5 a nonmetal for dispersion material is provided in the deposition crucible 7 so as to face the stationary substrate 2. The deposition source 15 is connected to an electric power source, not shown. As in the first embodiment, the deposition source 15, if being configured so that a rod-like source can 10 be supplied continuously, is effective in achieving mass production of composite material. Also, the stationary substrate 2 is formed of a metal for base material.

The chamber is provided with the atmospheric gas inlet 13 and the atmospheric gas outlet 14. From the atmospheric 15 gas inlet 13, an atmospheric gas produced by mixing a hydrocarbon gas such as acetylene gas and an inert gas such as argon gas is supplied, and the atmospheric gas is introduced in the chamber 1. Subsequently, the pressure in the chamber 1 is controlled to a predetermined value, and the 20 deposition source 15 is heated by causing an electric current to flow. Thereby, the metal for base material and the nonmetal for dispersion material are evaporated, and the evaporated particles are deposited on the stationary substrate 2 after passing through an acceleration probe electrode 16. At this time, the hydrocarbon gas such as 25 acetylene gas introduced in the chamber 1 decomposes into carbon and hydrogen as in the case shown in Fig. 6, and the carbon yielded by the decomposition is taken into the bulk

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body 6 formed on the stationary substrate 2 together with the evaporating metal for base material and nonmetal for dispersion material. Alternatively, the carbon reacts with the evaporating metal for base material or nonmetal for dispersion material to yield a stable carbide, and is taken into the bulk body 6 formed on the stationary substrate 2 in the carbide state. In this case, the quantity of the hydrocarbon gas in the atmospheric gas introduced in the chamber 1 or the heating temperature at the time of deposition is controlled, by which the carbon concentration in the bulk body 6 can be determined appropriately.

After the predetermined bulk body 6 is formed by the manufacturing method described with reference to Figs. 6 and 7, as in the case of the description of the first embodiment, the bulk body 6 is used as a single body, or it can be used as a composite material in which the concentration of dispersion material has been controlled by melting, mixing, and casting the bulk body 6 together with the metal for base material. Also, if necessary, the composite material can be rolled or heat-treated to control the crystal structure.

The following is a description of an example relating to the second embodiment.

Example 2: Example 2 represents a case where an aluminum-carbon composite material is manufactured by the sputtering method shown in Fig. 6. As an apparatus, a reactive magnetron sputtering apparatus was used, and as a sputtering target, a disk-shaped aluminum (purity: 99.999%) with a diameter of 203.2 mm and a thickness of 10 mm was used. Also, as a

stationary substrate, an aluminum (purity: 99.999%) foil with a thickness of 10  $\mu\text{m}$  was used.

A mixed gas of argon gas (purity: 99.999%) with a gas flow rate of 40 ccm and acetylene gas (purity: 99.5%) with 5 a gas flow rate of 20 ccm was supplied in the chamber, and the sputtering pressure was controlled so as to be 0.4 Pa. Electric power of 8 kW ( $24.7 \text{ W/cm}^2$ ) was applied to the aluminum target, and the substrate temperature was set at  $200^\circ\text{C}$ .

Sputtering was performed for 60 minutes, by which a bulk 10 body with a thickness of 80  $\mu\text{m}$  and a total weight of 6.14 g was formed on the stationary substrate. The carbon concentration in the formed bulk body was gas-analyzed, and it was found that 2.4 wt% of carbon was contained in the bulk body.

15 This bulk body was melted under vacuum together with aluminum (purity: 99.999%) which had been prepared separately, by which the aluminum-carbon composition was controlled so that the carbon concentration is 0.7% by weight, and casting was performed using a water-cooled copper mold. The 20 composite material formed by casting was observed under a microscope. As a result, it was verified that carbon with a particle diameter of about 1 mm was dispersed homogeneously into aluminum of base material in a form of  $\text{Al}\cdot\text{C}(\text{Al}_4\text{C}_3)$  phase as in the micrograph of Example 1 shown in Fig. 5.

25 The aluminum-carbon (0.7% by weight) composite material formed by casting as described above was formed into a sputtering target material, and an aluminum film was formed by using this target material under the same conditions as

those in Example 1. The film characteristics of the aluminum film was investigated, and resultantly it was verified that as in the case of Example 1, if the aluminum-carbon composite material obtained in Example 2 is used as a raw material for target material, a film having high hillock resistance and low electrical resistivity can be formed steadily.

#### INDUSTRIAL APPLICABILITY

As described above, according to the manufacturing method for a composite material in accordance with the present invention, a dispersion material can be dispersed homogeneously into a base material of a composite material, so that various composite materials can be manufactured generally independently of the composition of the composite material as compared with the conventional manufacturing method for a composite material. The composite material obtained by the manufacturing method in accordance with the present invention can meet the requirements for structural materials and electrode materials, and is suitable in each application because the dispersion material is dispersed very homogeneously into the base material and there is no internal defect such as a cavity or a cavity. In particular, if the composite material is used as a target material when wiring for a liquid crystal display or a semiconductor integrated circuit is formed, the required film characteristics can be realized steadily.

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